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# Adaptive Coating Technologies, LLC 4836 Morris Court Waunakee, WI 53597

Contractor's Progress, Status and Management Final Report for SBIR Topic No. A95-015, Proposal Title: Explosion Bonding of Titanium and Alloyed Steel

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## I. Phase I Project

### A. Introduction

Ultra high hardness steel is often used as an economical armor material, but its use as a structural material is limited because of its low ductility, high hardness, and difficulty in welding and machining. Titanium, however, with its high strength-to-weight ratio, is a good structural material with relatively high ballistic performance. Coupling titanium with an ultra high hardness steel could utilize the strengths of both materials and may provide an economical alternative for certain light armor applications such as the light armor vehicle (LAV).

Conventional welding of either titanium or high hardness steels presents various problems. For example, welding high hardness steels (i.e. Rockwell C60 minimum) demands careful attention because of the material's low ductility and extreme crack sensitivity. Dramatic microstructural differences among the weld metal, heat affected zone and steel base metal exist. These variances result in substantial ductility, hardness and strength differences, which may cause failure at the joint region. As a result, post heat treatments are usually required to relieve the material stresses. In addition, titanium requires special gas shielding techniques during welding to prevent surface oxidation. Using conventional welding processes to overlay titanium onto high hardness steels, or visa versa, would involve not only the problems previously mentioned but also the formation of various brittle intermetallics at the titanium-steel interface. The conventional welding processes therefore are highly impractical for attaining an adequate, cost effective titanium-steel bond.

Explosive bonding, however, could be more pragmatic, as it offers several advantages over the conventional welding processes for bonding titanium and steel. Explosive bonding of two materials generates an interface morphology that has minimal microstructural changes in the flyer and backer plates. Dilution between the two materials is minimized, and therefore brittle intermetallics, if present, are isolated and surrounded by ductile metal. Also with explosive bonding, the oxide layer of a material like titanium is removed just prior to bonding. The combination of reduced wetting and the intimate contact formed between the two unoxidized metals during explosive bonding should produce a joint that has high adhesive strength.

Commercial explosive bonding of a titanium flyer plate and steel backer plate is performed using steel with a surface hardness less than Rockwell C45 and a yield strength less than 100,000 psi. However, when backer materials have a hardness and yield strength of Rockwell C60 and 100,000 psi minimum, respectively, such as ultra high hardness steel substrates, explosive bonding is difficult even with the use of a low yield strength thin interlayer material.

Utilizing this information and innovative methods, this project produced composite titanium/steel plates that are extremely hard and exhibit minimal joint dilution and minimal intermetallic content. The explosive bonding created a waveless interface resulting in virtual elimination of the Ti-Fe intermetallics common with commercial

explosive bonding. After joining, the tool steel portion of the composite plate was heat treated using an infrared heating process to attain very high hardness without significantly effecting the titanium or the joint interface. Three plates were prepared using this methodology and submitted for ballistic testing.

### **B.** Summary

### 1. Explosive Bonding

Testing involved three titanium plates, grade 2 measuring 18" x 72" x.25", and three A-2 tool steel plates measuring 72" x 18" x .224". The titanium sheets were purchased from Tico Titanium, Farmington Hills, MI, and had a preheat treatment of 1300 F for 30 minutes. The tool steel plates were acquired from Houghton and Richards, Marlborough, MA. All material was drop shipped to New Mexico Tech.

The explosive bonding was conducted at New Mexico Tech-Energetic Materials Research & Testing Center in Socorro, N. M., under the guidance of Vasant Joshi. The tool steel plates were ground to remove any pitting. Table 1 is a summary of the explosive bonding work.

Table 1. Explosive bonding summary

Table 1. Explosive contains summary				
Plate Size	Part No.	Explosive Date	Comments	
9" x 9"	201P-07	3/4/96	Intermetallic region 0-20 microns	
18" x 30"	201P-08	3/11/96	Plates unbonded-defective prills	
9" x 9"	201P-06	3/21/96	Not ultrasonically tested	
18" x 18"	201P-01	3/22/96	2" x 2" unbonded	
18" x 18"	201P-02	3/22/96	50% unbonded	
18" x 18"	201P-03	4/11/96	No defects detected	
18" x 18"	201P-04	4/11/96	No defects detected	
18" x 18"	201P-05	4/11/96	No defects detected	
18" x 18"	201P-09	6/7/96	4 1/2" x 4 1/2" unbonded area	
18" x 18"	201P-10	6/7/96	5" x 10" unbonded area	

Initial explosive testing utilized (1) 9" x 9" tool steel and titanium plates. The composite plate formed, part no. 201P-07, was successfully joined using parameters determined in the MY1DL computer program. (Ref. 2) Upon metallographic examination of the composite plate, the titanium/tool steel interface exhibited minimal waviness with an intermetallic region. Figures 1 and 2 are photomicrographs showing the joint interface.

In an effort to eliminate the intermetallic region, the bonding parameters were altered. A second explosive shot was made using plates measuring 18" x 30" (Ref. Part no 201P-08). This attempt was unsuccessful, leaving the two plates unbonded and the tool steel plate cracked in several areas. This failure was due to defective ammonium nitrate/fuel oil prills. Subsequently, a new supplier of prill material was used for all future testing. Further study is required to determine the exact cause for the unbonded regions in the other plates.

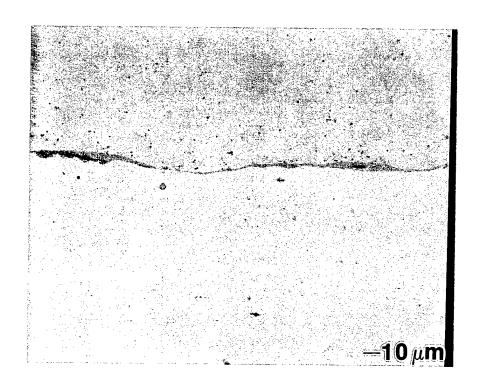


Figure 1. Photomicrograph of unetched hardened composite plate, specimen #1006. Note the two regions at the interface. The darker regions appear to be iron rich and the lighter regions appear to be titanium rich. 400X magnification

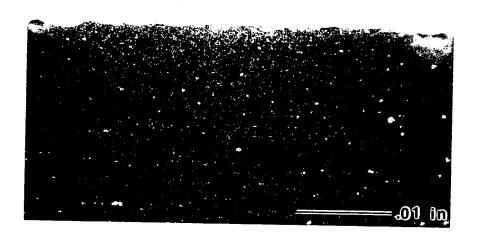


Figure 2. Photomicrograph of hardened composite plate, specimen #1006. Note the wave morphology at the interface. Material is etched with 3% nital. 100X magnification.

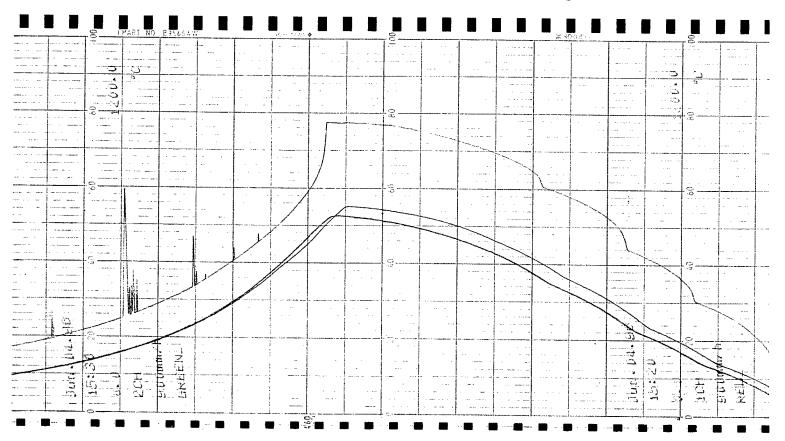
After bonding, the outer unbonded regions, approximately 2 inches around all four edges of the composite plates were sectioned using a handheld plasma cutting machine, and those pieces were scrapped. All plates were shipped to John Krebsbach, Adaptive Coating Technologies, Madison, Wis., for ultrasonic inspection and metallographic examination.

### 2. Heat Treating

After ultrasonic inspection, the tool steel portion of the composite plates was quenched and tempered in an infrared furnace. The heat treating was conducted at Oak Ridge National Laboratory in Oak Ridge, Tenn., under the guidance of Dr. Craig Blue. The goal of the hardening process was to achieve a minimum  $60~R_{\rm C}$  hardness through at least 1/2 of the tool steel thickness. The initial heating tests, using 1~3/4" x 1~1/8" x .429-434" thick bonded specimens removed from plate part no. 201P-07, were performed in a 4" diameter vacuum furnace. See table 3.

The hardening process started with embedding thermocouples at the titanium/tool steel interface and on the tool steel and titanium surfaces to study the thermal gradients under different heating conditions. To achieve a high hardness, the tool steel surface was heated to 950 C (1742 F), and that temperature was maintained for a minimum of 20 minutes. Likewise, a thermal gradient needed to maintain the tool steel/titanium interface at or below 600 C (1112 F) was held for 20 minutes. This gradient was maintained by placing the titanium side of the composite plate onto a water cooled copper fixture. A typical chart recording showing the temperature at each of the critical regions is shown in Figure 3.

Figure 3. Temperature Gradient Curves Provided by Infrared Heating



The blue curve illustrates the tool steel surface temperature, the red the interfacial temperature, and the green the temperature through approximately 1/2 of the titanium thickness. Full scale ordinate denotes a temperature of 1200 C, and the abscissa indicates a chart recording speed of 900 mm/hr.

After quenching from the hardening temperature, tempering tests were conducted on the hardened composite specimens at 205 C (400 F) for 2 and at 205 C for 4 hours. No thermal gradient was required for this process.

The effect of the interfacial debris on the thermal gradient exhibited in Figures 1 and 2 is unknown at this time. Table 2 identifies thermal conductivities of various titanium and iron rich materials at various temperatures. Based on this and the metallographic data, it appears that the interfacial condition/debris does aid in achieving a higher thermal gradient during infrared heating.

Table 2 Thermal Conductivities of Various Titanium and Iron Containing Materials

(W/cm·K) (Ref. 20 and 21)

Temperature	Iron	Titanium	TiO <sub>2</sub>	TiC
200 C (392 F)	.57	.1525	.0210	.2040
400 C (752 F)	.46	.1525	.0208	.1040
600 C (1112 F)	.35	.1025	.0207	.0640
900 C (1652 F)	.35	NA	.0208	.0540
950 C (1742 F)	.35	NA	.0208	.0440

(3) 12" x 12" plates, plate part nos. 201P-03 through 201P-05 were heat treated in a 27" x 24" infrared furnace completed in July '96. The hardening was performed at 970 -980 C (1778 -1796 F) for 20 minutes minimum. The plates were quenched using a water cooled fixture on the titanium surface and various flowrates of argon and/or helium gas passing over the tool steel surface. The resulting plates were then forwarded to TACOM for ballistic testing.

### 3. Inspection

Each plate larger than 9" x 9" was divided into five areas and ultrasonically inspected from the steel surface. Of particular importance was the magnitude of the wave reflected back from the joint interface. Results are presented in Table 1.

Before metallographic examination, each 1 3/4" x 1 1/8" x .429-434" thick asbonded and heat treated specimens were sectioned, mounted and polished. Figure 1 shows two different constituents at the interface: the darker region appears iron rich, while the lighter regions appears titanium rich. Also the joint interface has an atypical wavy interface compared to conventional explosive bonding parameters. Table 3 provides traceability information for the heat treat and metallographic specimens.

Table 3. Metallographic Specimen Information

Plate No.	Heat Treat Spec. #	Met. Specimen #	Comments
201P-07		1000	bonded specimen
201 <b>P-</b> 07	#3	1006	quenched specimen
201P-07	#4	1007	quenched specimen
201P-07	#1	1008	quenched and tempered specimen
201P-07	#2	1011	quenched and tempered specimen
201P-02	#5	1012	quenched and tempered specimen

The as-bonded specimen (#1000) primarily exhibits a ferrite and carbide structure. The hardened specimen (#1006) consists largely of martensite and some retained austenite. Figure 4 shows a representative area of etched tool steel microstructure at 1000X magnification in the as-quenched condition. Note the spheroidized carbide particles. Etchants used were 3% nital and a solution of sodium metabisulfite and distilled water.

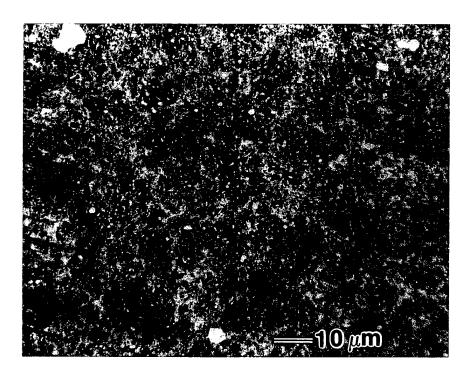


Figure 4. Representative area of etched tool steel microstructure, metallographic specimen #1006 in the hardened and as-quenched condition. 1000X. Etched using 3% nital solution.

Microhardness data was gathered on specimens at critical fabrication stages. Typical results are shown in Figure 5 and Table 4.



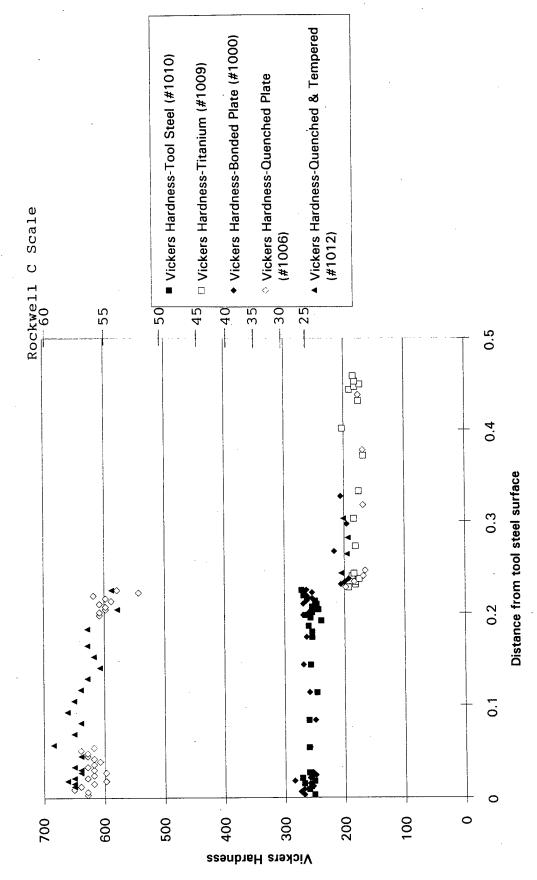


Figure 5. Microhardness data on specimens at critical fabrication stages.

Table 4. Microhardness values of Individual and Composite Plates

Fabrication Stage	Average Microhardness	Average Microhardness
	Tool Steel	Titanium
Tool steel as-received	257.6	
Titanium as-received		182.2
Explosive bonded plate	261.6	202.8
Hardened composite plate	607.6	175.4
Tempered composite plate	608.4	199.5

### C. Conclusions and technical substantiations

- 1) It is possible to create a waveless interface morphology between titanium and tool steel when explosive bonding titanium to A-2 tool steel material.
- 2) The bond strength of the waveless joint between the titanium and tool steel can withstand the thermal expansion of subsequent unidirectional hardening and tempering heat treatments without catastrophic failure. The composite material is also able to withstand repeated thermal cycling up to 950 C.
- 3) Infrared heating can harden tool steel very quickly. Normal hardening time at 950 C is 20-45 minutes, but with infrared heating, hardening occurs in less than a 20 minute hold time.
- 4) With infrared heating, a post heat treatment can attain a minimum of Rockwell C60 microhardness throughout more than 1/2 of the tool steel thickness without causing a significant deleterious effect on the titanium. Hardnesses of the tool steel climbed from an average 262 to 607 HV as a result of infrared heating. Likewise, the titanium dropped from 202 to 175 HV during the same cycle. Significantly, the composite plate was heat treated to produce an increase in hardness of the tool steel from 24  $R_c$  to 56  $R_c$ , a difference of 32  $R_c$ .
- 5) The infrared furnace allows for wide variability in heat treating parameters, such as heating rate, thermal gradients, holding times and quench rates. The thermal gradients between the tool steel and titanium are higher than any other known process.
- 6) An advantage over induction hardening, the infrared process can heat a material uniformly or unidirectionally. Induction heating requires a device to encompass the entire plate (i.e. induction coil); and subsequent induction heating heats the entire plate. Secondly, the distance between the infrared lamp and plate surface is not as critical as the distance between the induction coil and plate surface.
- 7) Considerable variability exists among commercially available ammonium nitrate/fuel oil pellets, or prills. Current quality control measures during prill manufacturing identifies the ratio and chemistry of the raw materials but not the morphology or detonation velocity of the final product.
- 8) Surface roughness of the plates prior to explosive bonding appears to be a critical factor in attaining adequate adhesion between flyer and backer plates.

Some reports state 125 micro finish or better is required. Nonetheless, all pitting should be eliminated.

# II. Detailed description of the analytical results

### A. Explosive Bonding

The chemistry of the as-received plates is shown in Table 5. The titanium was certified to meet the requirements of ASTM B-265 and ASME SB-265, grade 2; mechanical properties are shown in Table 6. The chemistry of the A-2 tool steel met recommended or commercial guidelines.

Table 5 Actual Chemical Composition of Titanium and Tool Steel Plates

Element	Titanium, Grade 2	A-2 Tool Steel
Iron	.10	Remainder
Oxygen	.16	
Nitrogen	.004	
Carbon	.02	
Hydrogen	13/14 ppm	
Titanium	Rem. (See note 1)	
Carbon		1.030
Silicon		.040
Manganese		.550
Sulfur		.013
Phosphorous		.024
Tungsten		.000
Chromium		5.170
Vanadium		.250
Nickel		.000
Molybdenum		.960
Cobalt		.000

Note 1: Residual elements (each) less than .10. Residual elements (total) less than .40.

Note 2: All values are weight percent unless otherwise specified.

Table 6- Mechanical Properties of As-Received Titanium Plate

Tensile data of Titanium	Longitudinal Test #1/#2	Transverse Test #1/#2
Tensile Strength (psi)	70400/71000	69400/71600
Yield Strength @ .2% offset (psi)	49900/50200	52700/54400
Elongation (%)	26/29	28/28

The tool steel and titanium plates were shipped directly to New Mexico Tech in Socorro, N.M. In the as-received condition, the tool steel plate surfaces exhibited areas of pitting, some severe. The plates were surface ground on the side that would become the joint, and the areas with excessive pitting were removed and

scrapped. The titanium plates were left in the as-received condition.

In all cases, the explosive bonding consisted of a titanium flyer plate and a tool steel backer plate with a 2" standoff between the plates. The tool steel plates had 2" wide momentum traps tack welded around the entire plate. A cardboard barrier was glued to the titanium flyer plate, and the two plates and cardboard barrier were then duct taped together.

At the explosion site, the taped assembly was placed on a mound of compacted sand. A Dupont Detasheet C4 strip was placed along the 22" plate width (18" plate plus 4" momentum trap widths), and the cardboard barrier was filled with ammonium nitrate fuel oil prills up to a height of 2.3"-2.5". This prill volume was calculated to yield a 2.2-2.7 km/sec detonation velocity. An ordnance personnel then placed the detonator in the center of the Detasheet, and ignited the charge using an RP 83 detonator. Plate part no. 201P-07 had slightly higher prill mass than previously stated.

The explosive bonding parameters, including the flyer plate velocity and position at impact, was accomplished using the MY1DL program. This program is a one dimensional Lagrangian hydrocode based on the difference equations as given by Neumann and Richtmyer<sup>1</sup>. The MY1DL program can calculate the time-distance-pressure and position during shock loading as well as energy input during passage of the shock wave. Shock waves are treated as a mathematical discontinuity, across which Rankine-Hugoniot relations are applied, and the concept of artificial viscosity makes it possible to calculate the steep increase in the pressures near the shock front. The various materials for flyer plate, impacted and compressed materials are discretized by cells, so that the basic equations can be substituted by finite difference elements. As the cell size becomes smaller, the finite differences approach the actual differences.

Lagrangian hydrocodes are generally simpler than Eularian hydrocodes, as they involve constitutive models. The MY1DL program uses the Mie-Gruneisen equation of state for the extension of the shock Hugoniot (experimental) and the thermal expansion at ambient pressure, into the adjacent regions in energy-pressure-volume (e-p-v) Equation of State (EOS) space. For the explosive, Gamma law approximations were used.

The input requires the material densities, shock Hugoniot coefficients (C and S), specific heats and the thermal expansion coefficients. The output for various cells gives pressure, temperature, energy and particle velocities. The program requires extensive knowledge of shock-wave theory and is reasonably accurate for estimation of pressure-time (p-t), particle velocity-time (u-t) and velocity-time (v-t) profiles.

### **B.** Heat Treating

Initial heat treating parameters were based on hardening, tempering and time-temperature transformation data of A-2 tool steel, as shown in Figures 6 through 8, respectively. Typical dimensional changes during hardening and tempering temperatures of A-2 tool steel and titanium are given in Table 7.

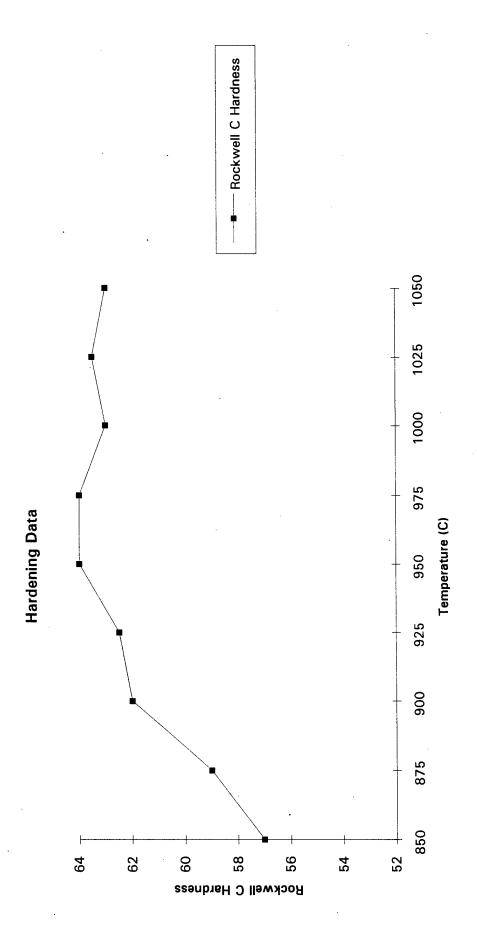


Figure 6. Hardening curve for A-2 tool steel as a function of temperature.

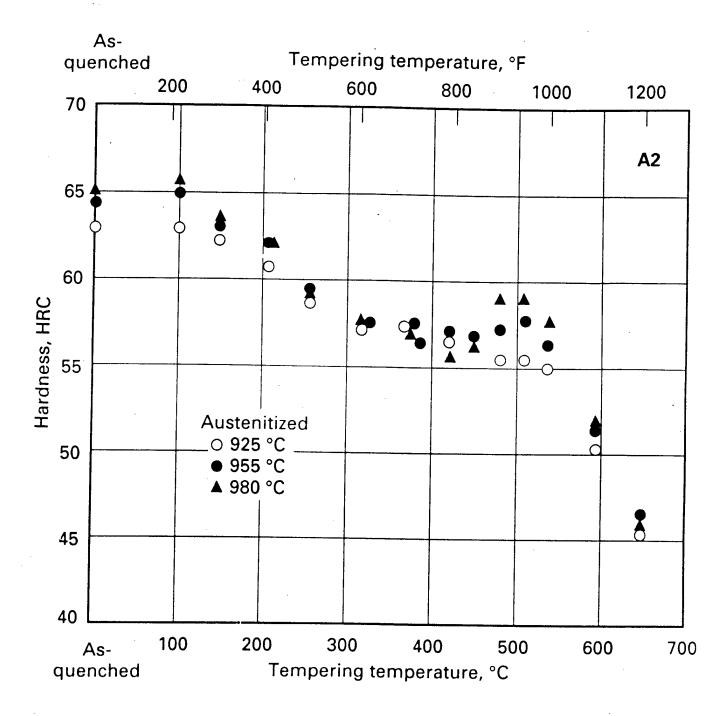


Figure 7. Tempering curve for A-2 tool steel as a function of temperature.

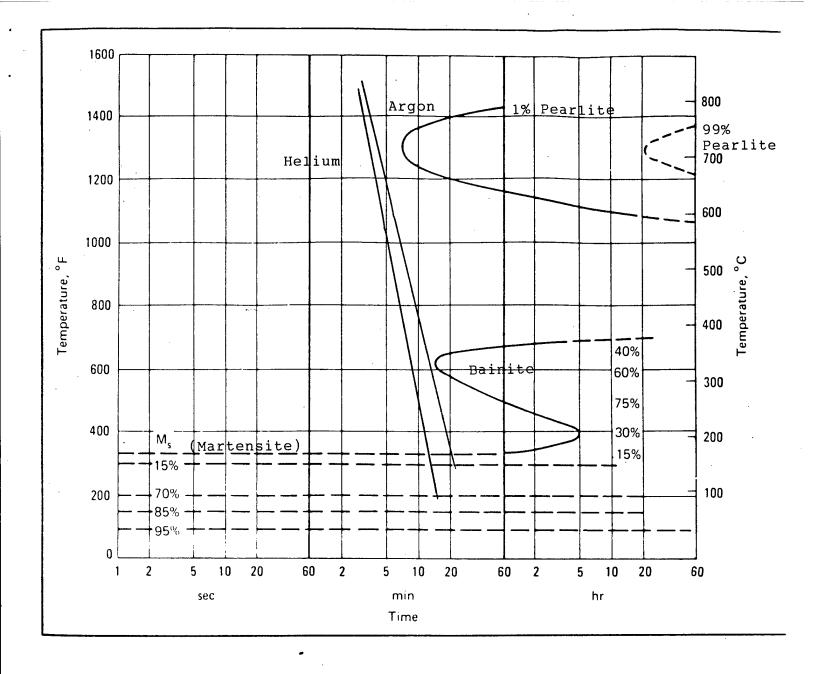


Figure 8. Time-temperature transformation curve for A-2 tool steel.

Table 7. Typical dimensional changes of tool steel and titanium during heating

Temperature	A-2 Tool Steel (total change	Titanium (ΔL/L <sub>0</sub> , %) Ref.
	in linear dimensions, %) Ref.	15
	16	
After air quench	.09	
300F temper	.06	.119
400F temper	.06	.170
500F temper	.08	.219
600F temper	.07	.275
700F temper		.340
800F temper	.05	.401
900F temper	.04	.460
1742F austenizing temp.	≈-2.36	.9893

Initially two 1 3/4" x 1 1/8" x .429-434" thick bonded specimens were machined to locate thermocouples to measure the titanium and tool steel surface temperatures and the joint interface temperature. The actual time-temperature data was recorded during infrared heating, and a chart showing the thermal gradient between the titanium and tool steel materials is shown in Figure 3. The total heating cycle, which is approximately 620 seconds (10.3 minutes), achieved a temperature at the tool steel surface of about 950 C for 50 seconds and a temperature at the interface of approximately 600 C, which prevents austenite formation and the associated contraction at the joint interface, a total gradient of 350 C. Note that the thermal gradient throughout 1/2 of the titanium thickness is only 24 C. Later testing showed that 20 minute hold times at 970-980 C hardening temperatures could be achieved as well as higher thermal gradients. All plates submitted for ballistic testing met this hardening time and temperature criteria.

This intrusive temperature recording methodology was time consuming and provided limited thermal gradient information. Later testing utilized thermocouples spotwelded to the side of the specimen. This method was less time consuming and yielded temperature approximations that were used throughout the remainder of the project.

After the proper hardening temperature/time was determined, the specimens were quenched by turning the infrared lamps off and by the water flow through the copper cooling fixture. Because the infrared lamps heat only the specimen and not the furnace walls, a faster quench occurred compared to conventional furnaces. The quenching process needed to drop the specimen temperature from the hardening temperature to 350 C (662 F) in less than 15 minutes to avoid bainite formation as shown in figure 8. No specimens were subjected to cryogenic cooling to achieve a higher martensitic percentage and the corresponding higher tool steel hardness values. Table 8 illustrates the thermal history for the plates submitted for ballistic testing.

Table 8 Composite Plates Submitted for Ballistic Testing

Plate No.	No of Heat Treat Cycles	Quenching Media	Quench Temperature/time
201P-03	1	Helium	975 C to 350 C in 8 min.
201P-04	2	Argon	980 C to 350 C in 17 min.
201P-05	2	Argon	980 C to 350 C in 16 min.

In all cases, tempering tests were accomplished after the specimen had cooled to room temperature. During the tempering cycle, no thermal gradient was established through the specimen thickness. To maintain a high tool steel hardness, the maximum tempering parameters were determined to be 205 C(400 F) for two hours. See figure 7. All plates submitted for ballistic testing met this tempering time and temperature criteria.

Initial infrared heating took place in a 4" diameter tube furnace using (6) 1500 watt tungsten filament lamps. The chamber was pumped down to 6 millitorr and then backfilled with an argon atmosphere. The bonded composite specimen was placed on a water cooled copper plate with a water inlet temperature of 18 C to maintain the desired thermal gradient. A silver paste between the composite plate and water cooled copper plate compensated for any distortion in the specimen caused by explosive bonding or heat treating.

After the small specimens were adequately quenched and tempered, the work was scaled up to quench and temper the 12" x 12" plates using a 27" x 24" vacuum furnace. The hardening process was accomplished in a similar way, except additional quenching was achieved on the tool steel surface using argon, helium or argon/helium gas mixtures. Typical quench rates are shown in figures 8 and 9. The tempering process of the larger plates involved no significant changes as compared to the small specimens.

Infrared energy is the portion of the electromagnetic spectrum between .78 and 1000 microns. The actual emission of a given source is dependent upon its temperature. Increasing the source temperature results in shorter overall wavelengths. The emissive energy is related to the following equation:

Q=KT<sup>4</sup>
Q-Total Emissive Power (watts/cm<sup>2</sup>)
K-Stefan Boltzmann Constant=5.56 x 10<sup>-12</sup>
T-Absolute Temperature (K)

Parameters of importance with high density infrared heating are defined in the following equation:

Q=(FV) x (ES) x (AT) x (K) x (TS<sup>4</sup> - TT<sup>4</sup>)
Q-Heat transfer between the source and target (watts/cm<sup>2</sup>)
FV-View factor between the source and target
ES-Emissivity factor of the source
AT-Absorption factor of the target
K-Stefan Boltzmann Constant

# Thermal Gradients During Infrared Heating

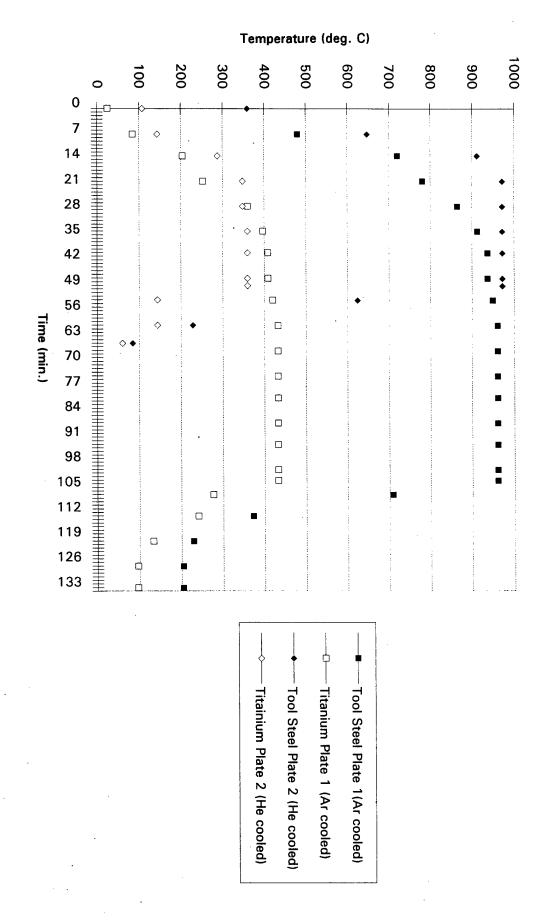


Figure 9. Temperature profiles within the composite plates using different quench media.

TS-Absolute Temperature of the source TT-Absolute Temperature of the target

The view factor term is the fraction between 0 and 1 that quantifies the amount of radiant energy emitted from the source that falls incident upon the target. The absorbed heat transfer (Q) results in a temperature rise of the target as defined by the following equation:

T=(Q) x (A) x (t)/(M)/(C<sub>p</sub>)
T-Product Temperature Rise (C)
A-Area of target (cm<sup>2</sup>)
t-Heating dwell time
M-Mass of the target
C<sub>p</sub>-Target specific heat (Watt-sec/kg-C)

### C. Inspection

Ultrasonic inspection of the 18" x 18" plates was conducted using a Quantum QFT 100 ultrasonic flaw detector and a C11, .5" diameter, 5 MHz transducer with a 0 degree impingement angle. A stainless steel wedge was used for calibration.

The metallographic specimens were sectioned from the corresponding heat treated 1 3/4" x 1 1/8" x .429-434" specimen using a Leco VC-50 low speed diamond saw. The specimens were cold mounted in epoxy, and the cured specimens were ground and diamond polished using a Struers DAP-V/Pedemin unit. Microhardness testing was performed with a Shimadzu Type M microhardness tester using either a 100 gm or 300 gm load for 30 seconds. Photomicrographs were taken using a Versamet microscope.

The intermetallic region evidenced in specimens taken from plate part no. 201P-07 varied in thickness from 0-20 microns as shown in Figure 1. Subsequent explosive bonding parameter changes resulted in a significant reduction and a greater uniformity of debris as shown in Figure 10.

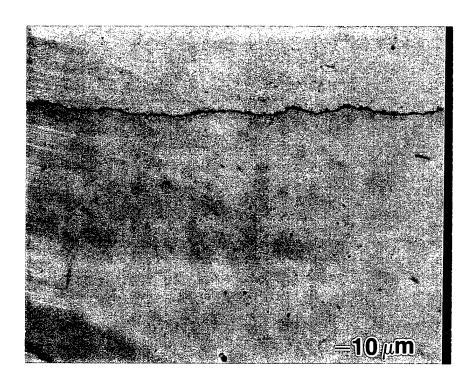


Figure 10. Joint interface of as-quenched and tempered specimen, #1012 showing uniformity of interfacial debris. 400X magnification in the as-polished condition.

In conclusion, this work demonstrates significant benefits to explosively bonding two materials coupled with infrared heating. This overall process may hold great promise for producing a steel/titanium composite armor plate.

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